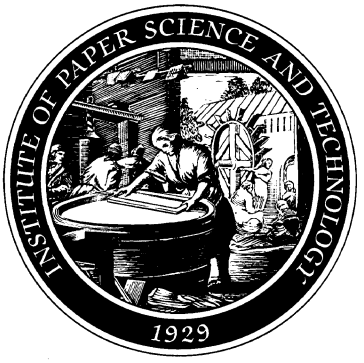

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The Behaviors of VOC's in Kraft Mills
Part I: Measurement Methods for the Quantification of the Contents and Vapor-Liquid Phase Partitioning of VOC's

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The Behaviors of VOC's in Kraft Mills

Part I: Measurement Methods for the Quantification of
the Contents and Vapor-Liquid Phase Partitioning of VOC's

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ABSTRACT

The understanding of VOC behaviours in Kraft mill streams is important to predict and control mill VOC emissions. Under the DOE sponsorship through the Agenda 2020 program, we conducted this series of a two-part study to understand VOC behaviours in Kraft mills: method development, VOC formation during pulping, and VOC vapor-liquid partitioning. In Part I of this study, we report on the analytical methods for the development of a database that describes both the contents and the vapor-liquid equilibrium partitioning of VOC's in various Kraft mill streams using a commercial headspace gas chromatography system. The methods are indirect, rapid, accurate, and automated. Preliminary measurements of VOC contents and Henry's constant of VOC's in various mill streams are conducted using the developed methods.

Application:

Analysis of VOC content and VLE partitioning in mill and environmental streams

Keywords:

VOC, Henry's law, vapor-liquid partitioning, headspace, GC, black liquor, mill streams.

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INTRODUCTION

With the increasingly restrictive environmental regulations, maintaining environmentally sound and technologically competitive operations in pulp and paper mills is the key to the success of the U.S. pulp and paper industry. The new toxic and permit provisions of the 1990 amendments require information on emissions of volatile organic compounds (VOC's) from pulp and paper mill sources. Many VOC's are now considered hazardous air pollutants (HAP's). Several studies on VOC emissions at Kraft mills have been conducted. Venketesh et al. [1] reported a millwide VOC prediction using a process simulation technique. The National Council of the Paper Industry for Air and Stream Improvement (NCASI) conducted a series of studies on VOC emissions at Kraft mills. NCASI's studies indicated that the release of VOC's during mill operations is determined by several factors [2]: (1) the VOC content in mill streams, (2) the fundamental thermodynamic vapor-liquid phase equilibrium behavior of the VOC's in mill streams, (3) the mass transfer associated with specific mill processes, and (4) the mill operating conditions, such as wood species, pulping chemicals used, water reuse in operation, etc. Some of the factors pertain to unit operating conditions and specific mill processes such as mass transfer in a unit operation. It is very difficult to generalize all the specific situations. However, the thermodynamic behavior of the VOC's should not depend on the characteristics of specific unit operations. Therefore, it is important to be able to measure VOC content in mill streams and understand the thermodynamic behavior of vapor-liquid phase equilibrium for computer model predictions.

It is not trivial to analyze samples from various mill streams using conventional direct analytical methods with delicate laboratory instruments through calibration because some of the mill streams are corrosive in nature. Gunshefki and Cloutier [3] developed a method to measure

MeOH contents in black liquor. However, their method modifies the sample matrix through the addition of chemicals to precipitate the solids in weak black liquors. The method has several disadvantages: (1) the amount of chemicals added (mass ratio of chemical over black liquor = 30:1) significantly dilutes the VOC concentration in the sample and reduces the measurement accuracy; (2) the method is only suitable for the analysis of weak black liquors as the solid precipitation method may not be used for other mill streams; and (3) the method is tedious, time-consuming, and not applicable for on-line analysis.

There are many methods available to study vapor-liquid phase equilibrium. Mackay and Shiu [4] presented a comprehensive review of the common methods to measure Henry's constants and their respective merits and deficiencies. Turner et al. [5] and Sherman et al. [6] separately presented their own comprehensive reviews of various methods for vapor-liquid equilibrium (VLE) studies—along with detailed comparisons of VLE data of aqueous organic systems obtained using these methods. Sherman et al. [6] also presented application limits of various available methods for VLE studies. The headspace gas chromatographic (HSGC) method gives a direct quantitative analysis of the vapor of a liquid sample matrix and therefore is very suitable for VLE studies. Much research on vapor-liquid phase equilibrium has been conducted using headspace GC systems [7-10]. The traditional HSGC method [7-10] for vapor-liquid equilibrium study requires quantitative determination of the equilibrium solute concentration both in the vapor and in the liquid phase through direct measurements using error-producing calibration procedures. To obtain experimental simplicity and high accuracy for practical applications, automated indirect HSGC methods will be desirable. Unfortunately, most of the existing indirect HSGC methods have practical difficulties to implement [11]. In this study, we report on the development of indirect methods for rapid, automated, and precise

determination of the contents and the vapor-liquid phase equilibrium partitioning of VOC's in mill streams using a commercial headspace gas chromatography system.

METHODOLOGY

Quantification of VOC Contents in Kraft Mill Streams

We developed an indirect measurement method for the quantification of VOC contents in liquids by headspace gas chromatography based on the thermodynamic vapor-liquid phase equilibrium. The method is schematically described in Fig. 1. We use two sample vials both filled with the same amount of sample solution. Then, we add a known small amount of concentrated solution into one of the vials. The volume of the solution added is very small compared with the volume of the original solution and therefore can be neglected. We conduct headspace GC analysis of each sample after a phase equilibrium was established within each vial. We can assume that the solute concentrations in these two sample vials are still very low or the solute concentrations are under infinite dilution, which is valid for most VOC's in mill streams even after the addition. Therefore, the solute VLE partitioning coefficients in these two vials are equal to the Henry's constant of the solute under consideration, which connects the two independent headspace measurements to determine the solute content in the original sample. The following is the derivation of the present HSGC method.

When a sample solution of volume V_l^0 with an unknown solute concentration of C_0 is introduced into a closed vial, the amount of solutes in the vapor at vapor-liquid phase equilibrium state can be described as:

$$n_1 = C_0 V_l^0 - \alpha C_1 V_l^0 = C_g^1 V_g^0, \qquad (1)$$

where C_1 and C_g^1 are the solute concentrations in the liquid and vapor under equilibrium, respectively, α is the solution volume expansion factor due to temperature change from state 0 to state 1, and V_g^0 is the headspace volume.

If a certain volume V_s of concentrated solution with a known solute concentration of C_s is added into this system, the existing equilibrium will be disturbed and a new equilibration state will be established after a while. The amount of solute in the vapor phase under the new equilibration state can be expressed as:

$$n_2 = C_0 V_l^0 + C_s V_s - \alpha C_2 V_l^0 = C_g^2 V_g^0, \tag{2}$$

where we assume that the total volume of the solution keeps the same as the volume of the concentrated solution added is negligible compared with the initial volume of the solution, i.e., $V_l^0 >> V_s$.

From Eqns. (1) and (2), we can obtain the concentrations of the solute in the liquid phase under the two equilibrium states,

$$\frac{C_1}{C_g^1} = \frac{C_0 V_l^0 - C_g^1 V_g^0}{\alpha V_l^0 C_g^1}, \text{ and} \tag{3}$$

$$\frac{C_2}{C_g^2} = \frac{C_0 V_l^0 + C_s V_s - C_g^2 V_g^0}{\alpha V_l^0 C_g^2}. \tag{4}$$

By assuming that the solute is infinite dilution in both vials, we can use the Henry's Law to connect Eqns. (3) and (4) as follows,

$$H_c = \frac{C_g^1}{C_1} = \frac{C_g^2}{C_2}. \tag{5}$$

Then, the initial solute concentration in the sample solution can be calculated from Eqns. (3)-(5) as,

$$C_0 = \frac{C_s V_s}{(C_g^2 / C_g^1 - 1)V_l^0}, \tag{6}$$

where, the ratio of the solute concentration in the vapor C_g^2/C_g^1 is proportional to the ratio r_A of the peak areas *A1* and *A2* of the solute detected from the two HSGC measurements. We can rewrite Eqn. (6) as,

$$C_0 = \frac{C_s V_s}{(A_2 / A_1 - 1)V_l^0} = \frac{C_s V_s}{(r_A - 1)V_l^0}. \tag{7}$$

Eqn. (7) can be used to determine the VOC contents in various mill streams. The advantage of the present method is that it does not require calibration.

Determination of VOC Henry's Constants in Kraft Mill Streams

The VOC's in mill streams are small quantities and can be treated under infinite dilution. Therefore, the Henry's law in Eqn. (5) best describes the VLE partitioning of VOC's in Kraft mill streams. Determination of Henry's constants is the key to understanding the thermodynamic VLE behavior of VOC's in mill streams.

The indirect GSGC method we developed uses two sample vials both filled with the same sample solution but with a significant variation in volume as schematically shown in Fig. 2. We conducted a headspace analysis of each sample after a phase equilibrium was established within each vial. The solute of the two systems has the same VLE Henry's constant as the two systems are identical, which can be used to connect the two independent headspace measurements to determine the solute concentration in the original sample. The derivation of the present method

is very simple. We can express the total moles M of the solute under equilibrium in the two vials using Eqns. (1) and (5),

$$M_1 = C_l^0 V_l^1 = C_l^1 V_l^1 + C_g^1 V_g^1 = C_g^1\left[\left(V_l^1 / H_c\right) + V_g^1\right], \tag{8}$$

$$M_2 = C_l^0 V_l^2 = C_l^2 V_l^2 + C_g^2 V_g^2 = C_g^2\left[\left(V_l^2 / H_c\right) + V_g^2\right], \tag{9}$$

where C_g is the concentration of solute in the vapor phase, and V_g is the vapor volume in the vial.

The dimensionless Henry's constant *Hc* can be derived from Eqns. (8) and (9),

$$H_c = \frac{V_l^1\left(1 - C_g^1 / C_g^2\right)}{C_g^1 / C_g^2\left(V_t - V_l^1\right) - V_l^1 / V_l^2\left(V_t - V_l^2\right)}. \tag{10}$$

The solute concentration in the vapor phase C_g is proportional to the peak area from GC measurement. Thus, we have

$$C_g^1 / C_g^2 = A_1 / A_2. \tag{11}$$

Substituting Eqn. (11) into (10), the dimensionless Henry's constant *Hc* can be determined

$$H_c = \frac{V_l^1\left(1 - A_1 / A_2\right)}{A_1 / A_2\left(V_t - V_l^1\right) - V_l^1 / V_l^2\left(V_t - V_l^2\right)} = \frac{V_l^1(1-r)}{r\left(V_t - V_l^1\right) - x\left(V_t - V_l^2\right)}, \tag{12}$$

where $r = A_1 / A_2$, and $x = V_l^1 / V_l^2$.

The approach that we took to derive the present indirect HSGC method is very similar to that of the Equilibrium Partitioning in Closed Systems (EPICS) method developed by Lincoff and Gossett [12]. However, the present method differs from the EPICS method significantly. It overcomes all the shortcomings of the EPICS method and has the following advantages: (1) it

does not require that one know the solute concentration to determine the VLE partitioning coefficient of the solution, which has significant importance for any industrial, environmental, and practical application; (2) it does not need to assume that the solute is under infinite dilution; therefore, the method is not only applicable to measure the Henry's constant, but also to measure the partitioning coefficient K of a solute in any solution; and (3) it has high precision even for determining a very small Henry's constant ($Hc<0.1$).

EXPERIMENTAL

Chemicals

We used methanol, methyl ethyl ketone (MEK), and acetone to mix with deionized water to make standard solutions of methanol-water, MEK-water, and acetone-water to validate the present methods.

We conducted measurements of VOC contents and VLE partitioning in various mill streams from four separate Kraft mills (Mills A, B, C, and D) to demonstrate the applicability of the present methods.

Apparatus and Operation

All measurements were carried out using an HP-7694 Automatic Headspace Sampler and Model HP-6890 capillary gas chromatography (Hewlett-Packard). A detailed description and the basic operation principles of the headspace sampler can be found in Dhasmana et al. [13]. GC conditions: HP-5 capillary column at 30°C; carrier gas helium flow (He): 3.8 mL/min. A flame ionization detector (FID) was employed with hydrogen and air flows of 35 and 400 mL/min, respectively. Headspace operating conditions: 25 minutes gentle shaking for

equilibration of the sample, vial pressurization time: 0.2 min, sample loop fill time: 1.0 min., and loop equilibration time: 0.05 min.

Temperature of the Headspace Sampler

For VOC content measurements, we set the temperature of the headspace sampler at 70°C to avoid water vaporization and obtain a good sensitivity as sufficient methanol will be present in the vapor phase at this temperature. The temperature was varied from 45-80°C for vapor-liquid partitioning measurements, close to the range of operating temperatures of most mill streams.

RESULTS AND DISCUSSION

VOC Content Measurements

Technique Validation

The experimental technique is validated using a set of standard VOC-water (methanol-water, MEK-water, and acetone-water) solutions with known concentrations. The present method measures the VOC concentration of the standard solutions according to Eqn. (7). The ranges of the concentrations of these three standard solutions are 100-2000, 10-100, and 1-10 ppm, respectively. The combination of these three concentration ranges covers trace species concentration over three orders of magnitude within the infinite dilution assumption limit. The comparison between the standard and the measured data is excellent. A perfect correlation between the standard and the measured is shown in Fig. 3. The repeatability of the method was demonstrated by using a standard methanol-water solution (methanol concentration = 800 ppm). A relative standard deviation of measured liquid methanol content is about 2.0% for the five measurements conducted, indicating that the repeatability of the technique is excellent.

Measurements of the VOC content in black liquor samples is very difficult because it has a complex composition such as dissolved solids and it is corrosive. We were able to separate various volatile species in black liquor using the GC system and the conditions stated previously [13]. We measured methanol contents in four black liquor samples from Mills A and B using the present method. Our data agree with the results obtained using the NCASI method [3] as shown in Table 1, indicating the applicability of the present method.

Measurements in Various Mill Streams

We measured methanol and MEK contents in various Kraft mill streams from Mill C using the present method. The results are shown in Table II. Mill C is a unbleached Kraft paper mill. For this particular mill, the data indicate: (1) the weak wash stream in the recovery cycle does not contain methanol; (2) the shower water and filtrate streams in the washers contain a significant amount of methanol; (3) the blow tank condensate stream from the digester also has a high content of methanol as indicated by the measurement of the sample from the hot water tank; (4) the white water from the paper-machine head tank for the present unbleached mill contains some methanol; (5) weak black liquor has a significant amount of methanol; and (6) MEK concentrations in various streams are in the order of a several hundred of ppb, and they do not have a direct correlation with the concentrations of methanol. The measured methanol contents in various streams are reasonable with practical knowledge. Similar conclusions can be drawn from the measurements of VOC concentrations of various streams in another unbleached Mill D as shown in Table III . The absolute concentrations of VOC's in mill streams depend on various factors such as water dilution, mill operating conditions, and VOC formation during pulping that will be reported in the future.

Henry's Constant Measurements

Technique Validation

We found from mathematical precision analysis [11] that the present method can be very accurate by properly choosing the two key experimental parameters, i.e., the solution volume ratio x and the volume of the sample V_l^1 or V_l^2. We applied the present method to measure the methanol Henry's constant in a methanol-water mixture. The Henry's constant of methanol in water under a temperature range of 45-80°C is very small (<0.005); therefore, it is a good precision test of the present method. The data obtained using the present indirect HSGC method show excellent agreement with those in the literature [14-18] as shown in Fig. 4. A linear regression analysis shows that the logarithm of all the data fit to a straight line with the inverse of temperature very well. The linear relationship agrees with thermodynamic theory, i.e., the Henry's constant is related to the partial molar excess enthalpy, which is a weak function of temperature. The data demonstrate the validity and the accuracy of the present method.

Measurements in Black Liquors

We applied the present method to measure Henry's constant of methanol in black liquor. Both softwood and hardwood black liquor samples from Mill B were used in this study. Fig. 5 shows the effect of temperature on the methanol Henry's constant in black liquors along with the data obtained in the methanol-water mixture. The results indicate that the logarithm of Henry's constant in the two black liquor samples decreases linearly with the inverse of temperature. It appears that the slops of the three sets of data presented are the same; the differences are within the error margin. However, the variation in the measured Henry's constant among these three types of samples is very significant. It is obvious that there is a significant variation in the

composition, ionic strength, solid contents, etc., among these samples, which may cause the large variation in the Henry's constant. We are conducting a detailed study to understand the effect of various parameters on the Henry's constant, which will be reported in Part II of this study.

CONCLUSIONS

The present study reported on the development of indirect methods for measuring VOC concentration and vapor-liquid phase equilibrium partitioning coefficients in mill streams using a commercial headspace gas chromatography system. The methods are rapid, automated, and accurate, and do not require calibration and modification of the sample matrix. The method can also be applied to various industrial and environmental streams. Preliminary measurements of VOC content and Henry's constant of methanol in Kraft mill streams using the methods developed were conducted. Significant variation in the measured data was observed, indicating the complicated nature of the behaviors of VOC in Kraft mill streams.

ACKNOWLEDGEMENT

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18. Lebert, A. and Richon, D.J., J. Agric. Food Chem. 32 (1984) 1156.

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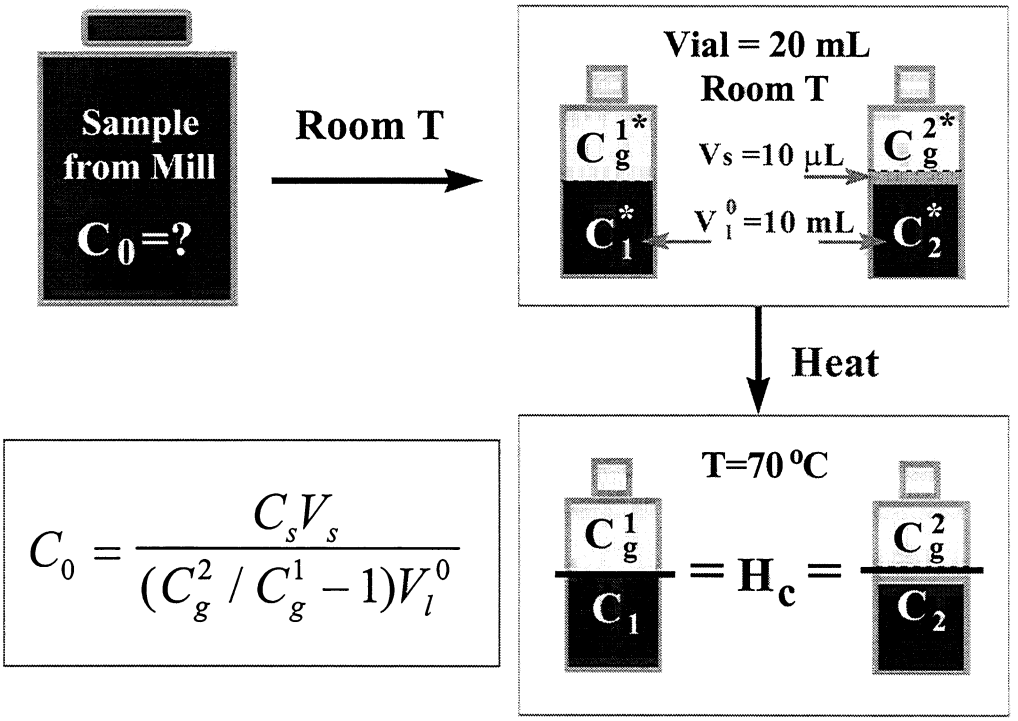
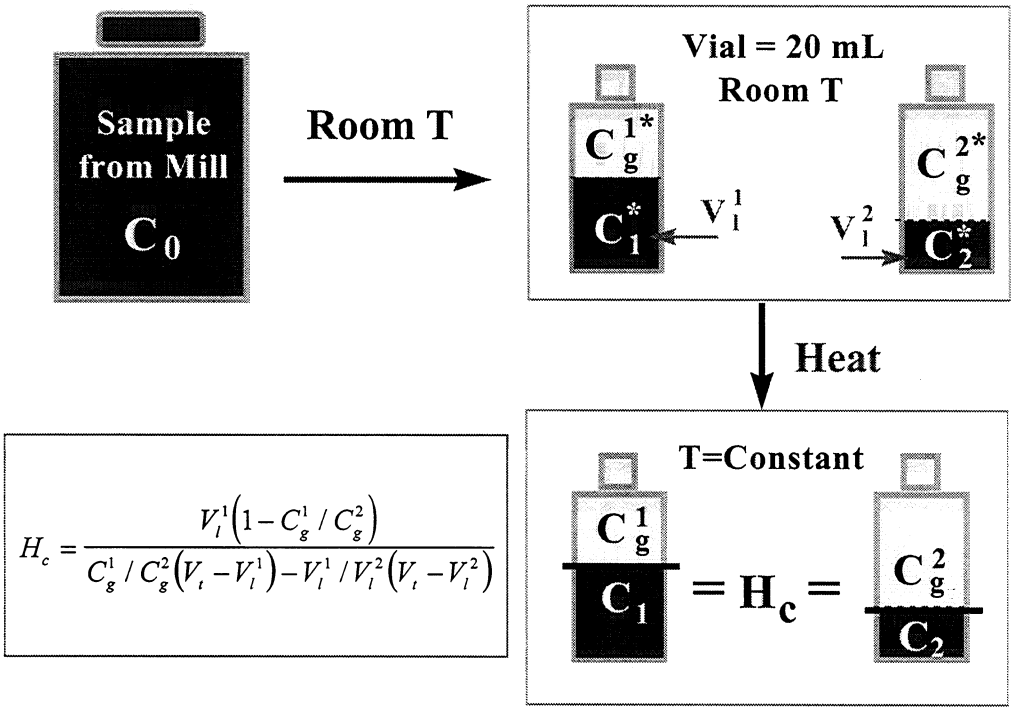
Fig. 1



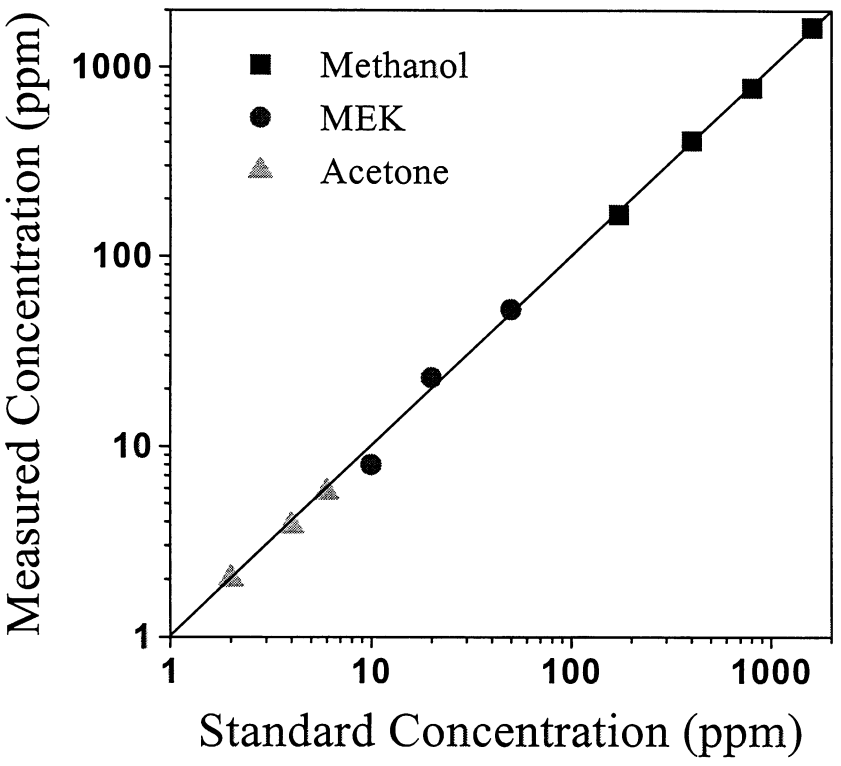
$$C_0 = \frac{C_s V_s}{(C_g^2 / C_g^1 - 1) V_l^0}$$

Fig. 2



$$H_c = \frac{V_l^1\left(1 - C_g^1 / C_g^2\right)}{C_g^1 / C_g^2\left(V_t - V_l^1\right) - V_l^1 / V_l^2\left(V_t - V_l^2\right)}$$

Fig. 3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100
10
1
Methanol
MEK
Acetone
1
10
100
1000
Standard Concentration (ppm)

Fig. 4

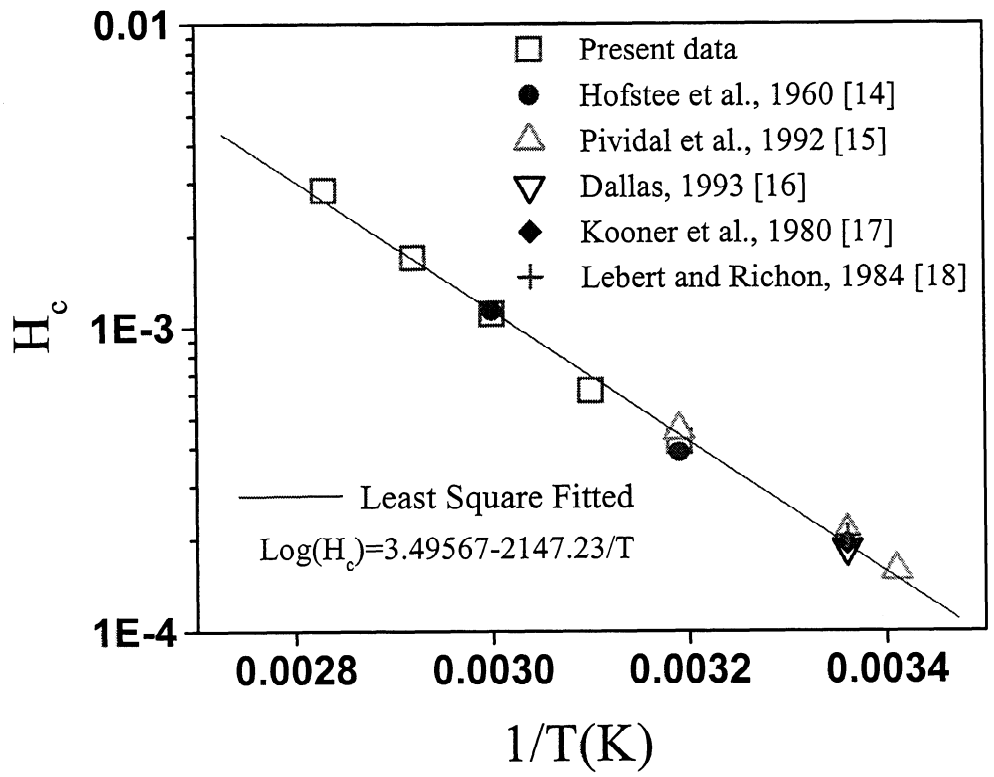
0.01
1E-3
1E-4
H_c
0.0028
0.0030
0.0032
0.0034
1/T(K)
Present data
Hofstee et al., 1960 [14]
Pividal et al., 1992 [15]
Dallas, 1993 [16]
Kooner et al., 1980 [17]
Lebert and Richon, 1984 [18]
Least Square Fitted
Log(H_c)=3.49567-2147.23/T

Fig. 5

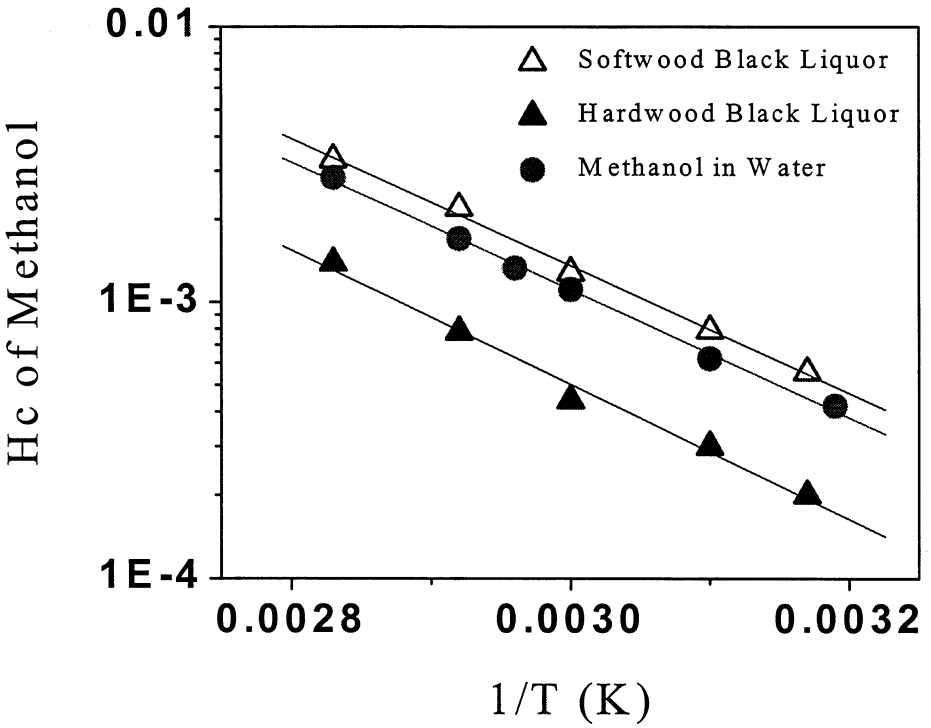
0.01
1E-3
1E-4
Hc of Methanol
0.0028
0.0030
0.0032
1/T (K)
Softwood Black Liquor
Hardwood Black Liquor
Methanol in Water

Table I

SAMPLE	Solids Content (%)	Methanol Concentration (ppm)		
		NCASI Method	Present Method	Difference (%)
Softwood, Mill A	15.2	775	736	5.0
Hardwood, Mill A	17.1	961	906	5.7
Softwood, Mill B	11.5	434	419	3.5
Hardwood, Mill B	10.8	527	560	-6.3

Table II

Sample Number	Sample Location Description	Methanol (ppm)	MEK (ppb)
1	weak wash liquor from recovery cycle	<1	74.5
2	white water from paper-machine head tank	40	0
3	pulp wash shower water first stage digester (D1)	277	165
4	filtrated stream from first washing stage (D1)	251	433
5	filtrated stream from second washing stage (D1)	238	316
6	filtrated stream from first washing stage (D2)	201	583
7	filtrated stream from second washing stage (D2)	172	481
8	condensated stream of blow tank steam in hot water tank	315	251
9	weak black liquor to evaporator	272	307

Table III

Sample Location Description	Label	MeOH	Acetone	MEK
		(ppm)	(ppb)	(ppb)
1st stage showers, 2nd stage filtrate	No. 1 Washer	209.5	494	406
	No. 2 Washer	275.0	484	394
2nd stage showers, 1st stage filtrate	No. 1 Washer	149.8	980	439
	No. 2 Washer	312.7	852	511
3rd stage showers	No. 1 Washer	122.6	683	452
	No. 2 Washer	203.8	715	526
side combined condensate (blow recovery)	No. 1 Washer	178.6	1327	592
	No. 2 Washer	382.6	1113	774
1st stage filtrate	No. 2 Washer	349.0	520	270
3rd stage filtrate	No. 1 Washer	332.2	930	564
combined weak black liquor to recovery		292.4	670	315
evaporator seal tank condensate	No. 2 (M24-0542)	93.1	762	665
	No. 2 (24-0529)	673.1	4138	425
evaporator clean condensate	No. 3	2.0	-	31
	No. 3 (24-0509)	1.7	-	231
evaporator	No. 2	27.1	-	218
evaporator seal tank	No. 1 (M24-0027)	3954.4	11	2706
combined condensate of hotwell	No. 3	659.1	16067	7826
paper-machine condensate	No. 1	3.3	-	59
	No. 2 & No. 3	3.5	-	124
top headbox	No. 1	57.4	323	108
	No. 2	55.3	260	129
base headbox	No. 1	88.8	433	167
	No. 2	64.9	315	158
headbox	No. 3	46.8	-	-
wire pit	No. 1	86.5	375	141
	No. 2	91.8	508	129
	No. 3	90.9	-	-
water reclaim sump	No. 1	13.0	192	62
vacuum dump	No. 2	20.6	-	-
side hill screen drain off	No. 3	65.7	-	-